

This Page Is Inserted by IFW Operations
and is not a part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

IMAGES ARE BEST AVAILABLE COPY.

**As rescanning documents *will not* correct images,
please do not report the images to the
Image Problem Mailbox.**

PATENT SPECIFICATION

DRAWINGS ATTACHED

Inventor: JOHN LEYLAND

L080.863

L080.863



Date of filing Complete Specification: May 14, 1964.

Application Date: Feb. 22, 1963.

No. 7191/63.

Complete Specification Published: Aug. 23, 1967.

© Crown Copyright 1967.

Index at acceptance: —B1 F4F; B1 X20; C2 C(3C7, 3C10)

Int. Cl.: —B 01 j 1/00

COMPLETE SPECIFICATION

Chemically Reacting Gases with Liquids

We, UNILEVER LIMITED, a Company registered under the laws of Great Britain, of Port Sunlight, in the County of Chester, England, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to processes and apparatus for chemically reacting a gas with a liquid.

In many reactions between gases and liquids, such as in chlorination, sulphonation and sulphonation processes, the action between the gas and the liquid phases is effective only when the gas and liquid molecules are brought into intimate contact, and the exothermic heat generated, or the endothermic heat required, is removed from or added to the mixture of reactants to control the rate of reaction or to bring it to completion. It is with processes of this kind that the present invention is concerned.

According to the invention there is provided a process for chemically reacting a gas with a liquid, which process comprises: feeding the liquid reactant on to one surface of a rapidly rotating disc of a heat-conductive material so that the liquid reactant is spread across that surface in a fast moving thin film by centrifugal forces arising from the motion imparted to the liquid reactant by the frictional drag of the disc; exposing the liquid film to the gaseous reactant to cause the gas and the liquid to react with one another; and, as the film flows across the disc of heat-conductive material, removing the exothermic heat of reaction from, or supplying the endothermic heat of reaction to, the liquid film through the disc of the heat-conductive material.

Such a process has the advantage that it provides not only a very short residence time, a very thin film of liquid reactant, and

despite the thinness of this film, a remarkably high throughput of commercial significance, but also minimises back-mixing of the reaction product with unreacted material, thus helping the reaction to go to completion and reducing the opportunity for unwanted subsequent reactions to occur. Moreover, as the fast moving thin film is caused to expand radially as it progresses across the surface of the disc, the liquid at the gas-liquid interface is continually replaced so that the concentration of molecules of reaction product at the interface is dispersed immediately and further liquid reactant molecules are presented at the interface for reaction with the gas.

The invention also provides apparatus for chemically reacting a gas with a liquid, which comprises a rotatable disc of a heat-conductive material, means for feeding a liquid reactant to the surface of one side of the disc and adjacent to its centre, so that when the disc is rotated the liquid reactant is spread across that surface in a fast moving thin film by centrifugal forces arising from the motion imparted to the liquid reactant by the frictional drag of the disc; means for feeding a gaseous reactant to the space adjacent to the surface of the disc on which the liquid film is formed for exposure of the liquid film to the gaseous reactant, heat transfer means for removing heat from or supplying heat to the surface of the opposite side of the disc, and means for collecting the reaction product from the periphery of the disc.

In order to make the most effective use of the frictional drag forces, the surfaces of the rotatable disc should be substantially planar. The disc in use can, for example, be rotated at 1540 revolutions per minute. The material of construction of the rotatable disc should be one of good thermal conductivity, for example mild steel, aluminium, copper or silver, and those components in

[P]

contact with reactants should be of corrosion resistant material.

5 Preferably the liquid reactant is fed on to the disc over the whole of an area of the surface of the disc surrounding the centre of the disc so that the most efficient use is made of the disc surface, and the means for feeding the liquid reactant to the disc is arranged accordingly. The way in which the liquid is fed on to the rotating disc will influence the properties of the film formed and the performance of the apparatus. The liquid can advantageously be fed on to the disc in an even manner by providing a feed box at the end of the liquid feed pipe, such that a circular orifice, centred on the axis of the disc, is produced between the feed box and the disc surface. The liquid reactant is conveniently fed on to the disc through a passage in a stationary shaft arranged co-axially with the rotating disc.

20 Preferably the flow of gaseous reactant is concurrent with the flow of liquid reactant, but countercurrent flow can be employed in circumstances where there is advantage or no disadvantage in allowing fresh gas to contact liquid reactant containing a large proportion of reaction product, and it is desired to help the reaction to go to completion. The gaseous reactant can if desired be under superatmospheric pressure or reduced pressure: it can be fed through a passage in a stationary shaft arranged co-axially with the rotating disc, preferably also one which carries the liquid reactant feed in a separate passage. Its velocity of entry should not be so great as to strip the liquid film off the rotating disc. The gaseous reactant can suitably be exposed to the film of liquid reactant in a chamber defined by the rotating disc and a casing attached to the periphery of the disc, the casing being rotatably mounted on the stationary shaft.

45 A stationary disc is preferably provided parallel to and slightly displaced from, the rotating disc, so as to confine the path of the gaseous reactant to the surface of the film of liquid reactant, the gaseous reactant being fed evenly into the space between the two discs, preferably at a point adjacent to the liquid feed. The presence of the stationary disc not only ensures flow of gas parallel to the liquid on the rotating disc surface, but also increases turbulence in the gas at the gas/liquid interface, and thus improves the mass transfer performance between the gas and liquid.

60 In some instances heating of the rotating disc surface opposite that on which the liquid reactant flows can be by means of radiation or convection type heating, and cooling can be by means of air or other gases, but the exothermic heat of reaction is preferably removed from, or the endothermic heat of reaction supplied to, the liquid film by causing

a film of a heat transfer liquid to flow across the surface of the side of the rotating disc opposite that across which the liquid reactant flows, the heat transfer liquid flowing across the disc surface by centrifugal forces arising from the motion imparted to it by the frictional drag of the disc. Advantageously such heat transfer liquid is fed on to the opposite side of the disc over the whole of an area of that surface surrounding the centre of the disc. When considering provision of rapid heat exchange with the reactant liquid it is not merely sufficient to obtain good heat transfer between the reacting liquid and the heat-conductive material, and an equally good heat exchange performance between the heat transfer liquid and the heat-conductive material is necessary. A particular advantage of carrying out the process using a film of liquid heat transfer material as described above is that the heat exchange advantages obtained by using a highly turbulent thin liquid film are achieved on both sides of the heat-conductive material.

Accordingly in apparatus of the invention the heat transfer means preferably comprises means for feeding a heat transfer liquid to the surface of the opposite side of the rotatable disc and adjacent to its centre, so that when this disc is rotated the heat transfer liquid is spread across that surface in a fast moving thin film by centrifugal forces arising from the motion imparted to the heat transfer liquid by the frictional drag of the disc, and the means for feeding the heat transfer liquid to the rotatable disc is preferably arranged for the feeding of liquid over the whole of an area of the surface of the disc surrounding the centre of the disc.

The reaction product can be collected from the periphery of the disc, for instance by a dip pipe, and preferably from an annular channel at the periphery of the disc by a pipe whose inlet is within the channel, and made to flow through a passage in a stationary shaft arranged co-axially with the rotating disc which carries other passages through which the gaseous and liquid reactants are fed to the disc. A separate gas outlet from the reactor can be provided such as by a separate channel in the stationary shaft. This prevents further reaction between gas and liquid from occurring after leaving the rotating disc. This arrangement will normally only be used when a stationary disc as described above is employed, as a stationary disc would then prevent the gas escaping from the reactor before it contacted the liquid film. The stationary disc can contain channels leading at one end to the periphery of the disc and connecting at the other end with the discharge channel through the stationary shaft, the whole being designed so that the reaction product is removed from the periphery of the disc through these channels rather than through a

dip pipe. This arrangement provides a more even removal of liquid and reduces the width of the casing required.

- Depending on the rate of reaction and the heat of reaction the liquid reactant can be passed through two or more units of apparatus of the invention in series. Fresh gaseous reactant can be fed to each unit or the gas can pass through the series of units. Different liquid temperatures can be adopted in the separate units, for instance to minimise side reactions.

- A process of the invention is particularly advantageous for carrying out sulphonation or sulphation reactions in which sulphur trioxide or a mixture of sulphur trioxide and air or an inert gas is reacted with organic compounds, for example, hydrocarbons or fatty alcohols.

- Examples of hydrocarbons that can be sulphonated using the process of the invention are the aromatic and aliphatic-aromatic hydrocarbons, especially those alkylbenzenes which on sulphonation give rise to products having surface-active properties. Examples of aliphatic-aromatic hydrocarbons are the alkylated benzenes having 9—18 carbon atoms in the alkyl group. Other sulphonation reactions that can be carried out are those for sulphonating C_8 — C_{22} fatty acids to give alpha sulphonyl fatty acids.

- Fatty alcohols which are readily sulphated in accordance with a process of the invention are, for example, the straight chain alcohols containing 8—20, preferably 12—18, carbon atoms. Ethylene oxide derivatives such as the alkylphenol-ethylene oxide condensates, for example, containing 9—12 carbon atoms in the alkyl group, or ethylene oxide derivatives of C_8 — C_{22} fatty alcohols, can also be sulphated using a process of the present invention.

- Examples of other reactions between gases and liquids in which a process of the invention can be applied are the chlorination of alkali for the production of hypochlorite, the hydrogenation of edible oils, and the production of amides by reacting ammonia with fatty acids.

- The gaseous reactant can be the vapour of a material which is liquid under normal atmospheric pressure or temperature. The liquid reactant can be a material which is normally a solid but which has been liquefied by heating or obtained in liquid form by dissolution in a suitable solvent. Mixtures of gases or mixtures of liquid reactants can also be used.

- An embodiment of the invention will now be described with reference to the accompanying drawing.

- Referring to the drawing, a disc 1 of heat-conductive material is joined at its periphery to a casing 2 mounted on a bearing housing 3 which rotates on bearings 4, 5 mounted

on a hollow stationary shaft 6. Between the casing 2 and the shaft 6 is arranged a sealing gland 7.

In the shaft 6 are accommodated two steel tubes 8, 9 providing inlet passages 10, 11 and an outlet passage 12. Tube 9 terminates in port 13, the free edge 14 of which is spaced by a narrow gap 15 from the surface 16 of disc 1. Passage 10 communicates through port 17 at the end of tube 8 with a space 18 between the disc surface 16 and casing 2. Passage 12 between shaft 6 and tube 8 is in communication with the inlet of a pipe 19 which extends downwards through space 18 to an annular channel 20 defined by the periphery of disc 1 and casing 2.

Disc 1 is extended at its periphery on the side farther from casing 2 to form a flange 21 which is shaped to provide an annular gutter 22. Close to the surface 23 on the reverse side of disc 1 is arranged a liquid feed box 24 over the end of which is slidably mounted an adjustable sleeve 25, the free edge 26 of which is spaced by a narrow gap 27 from the surface 23 of disc 1. The liquid feed box 24 is provided with an inlet 28, and an outlet pipe 29 extends into the annular gutter 22.

In operation, the disc 1 is rotated at high speed by suitable means (not shown) such as by an electric motor connected by belt and pulley to housing 3. Liquid reactant A is fed along channel 11 in tube 9 and is under pressure so that at port 13 there is maintained a body of the liquid reactant A in contact with the disc surface 16 over an area surrounding the centre of the disc. The disc 1 rotated at high speed draws the liquid A out through the narrow gap 15 between the edge 14 of tube 9 and surface 16 and causes it to flow across the surface 16 of disc 1 as a thin, fast moving film under the influence of centrifugal forces arising from the motion imparted to the liquid by the frictional drag of the disc.

The gaseous reactant B is fed along the passage 10 between tubes 8 and 9 and is passed out through port 17 into the space 18 between disc 1 and casing 2. Here the gas which is now exposed to the liquid film moving over the disc surface 16 reacts with the liquid A. A very rapid and complete reaction is possible due to the intimate and substantial contact between the molecules of the gas and those of the fast moving liquid film.

The liquid reaction product C on reaching the periphery of disc 1 flows circumferentially around the annular channel 20, is collected by pipe 19, and passes with considerable kinetic energy along it and through passage 12 between tube 8 and shaft 6. Any unreacted gas is removed with the reaction product C.

The heat of reaction is withdrawn from, or (where the reaction is endothermic) supplied

to, the reaction mixture by causing a heat transfer liquid D to flow on the surface 23 of disc 1. This heat transfer liquid is fed through inlet pipe 28 into feed box 24 to maintain a body of liquid in contact with the surface 23 of the disc 1 over an area surrounding the centre of the disc. The heat transfer liquid is drawn out through gap 27 between the edge of sleeve 26 and surface 23 of disc 1 and flows rapidly as a thin film over surface 23. An immediate and efficient heat transfer between the two liquids flowing over the surfaces 16 and 23 of the disc 1 is obtained. The heat transfer liquid D on reaching the edge of disc 1 flows circumferentially in the annular gutter 22 and passes out through outlet pipe 29 with considerable kinetic energy.

The drawing also shows as an optional feature a stationary disc 30 attached to port 17 and extending through space 18 parallel to disc 1, leaving a gap 31 between its periphery and casing 2 for passage of the reaction product to channel 20 and pipe 19.

The following Example illustrates the use of the apparatus described above without the stationary disc 30.

EXAMPLE

This Example concerns the sulphomation of a commercial alkylbenzene hydrocarbon of average molecular weight 245, whose alkyl portion is essentially straight chain secondary alkyl, using a mixture of sulphur trioxide vapour and air as sulphonating medium. The disc 1 had a diameter of 12½ inches and a thickness of 3/16 inch, was constructed of mild steel, and was rotated at a speed of 1,540 revolutions per minute. The hydrocarbon was supplied at 20° C. and at a rate of 90 lb./hr. through pipe 9 of ½ inch diameter, the gap 15 between the end of the pipe and the disc surface 16 being 1/32 inch. Cooling water at 20° C. was supplied in a similar manner to the reverse side 23 of the disc at a rate of 2,000 lb./hr. A mixture of sulphur trioxide vapour and dry air in the respective proportions of 1:2 by volume was supplied through passage 10 at a rate of 390 cuft./hr. (at S.T.P.) Sulphonation occurred on the disc surface 16 and the material left the apparatus at a temperature in excess of 100° C. and was found on analysis to contain 93.7% of alkylaryl sulphonate together with unreacted alkylaryl hydrocarbons. The material obtained from the apparatus was as acceptable in respect of colour as that obtained from a conventional batch stirred-tank reactor.

WHAT WE CLAIM IS:—

1. A process for chemically reacting a gas with a liquid, which process comprises: feeding the liquid reactant on to one surface of a rapidly rotating disc of a heat-conductive

material so that the liquid reactant is spread across that surface in a fast moving thin film by centrifugal forces arising from the motion imparted to the liquid reactant by the frictional drag of the disc; exposing the liquid film to the gaseous reactant to cause the gas and the liquid to react with one another; and, as the film flows across the disc of heat-conductive material, removing the exothermic heat of reaction from, or supplying the endothermic heat of reaction to, the liquid film through the disc of heat-conductive material.

2. A process as claimed in Claim 1, in which the liquid reactant is fed on to the rotating disc over the whole of an area of the surface of the disc surrounding the centre of the disc.

3. A process as claimed in Claim 1 or Claim 2, in which the exothermic heat of reaction is removed from, or the endothermic heat of reaction supplied to, the liquid film by causing a film of a heat transfer liquid to flow across the side of the rotating disc opposite that across which the liquid reactant flows.

4. A process as claimed in Claim 3, in which the heat transfer liquid is fed on to the rotating disc over the whole of an area of that surface surrounding the centre of the disc.

5. A process as claimed in any preceding claim, in which the reaction product is collected from the periphery of the rotating disc.

6. A process according to Claim 5, in which the reaction product is collected from an annular channel at the periphery of the rotating disc by a pipe whose inlet is within the channel.

7. A process as claimed in any preceding claim, in which the liquid reactant is fed on to the rotating disc through a passage in a stationary shaft arranged co-axially with the rotating disc.

8. A process as claimed in Claim 7, in which the gaseous reactant is also fed through a passage in the stationary shaft.

9. A process as claimed in Claim 7 or Claim 8, in which the gaseous reactant is exposed to the film of liquid reactant in a chamber defined by the rotating disc and a casing attached to the periphery of the disc, the casing being rotatably mounted on the stationary shaft.

10. A process as claimed in any one of Claims 7 to 9, in which the reaction product is collected from the periphery of the rotating disc and flows through a passage in the stationary shaft.

11. A process as claimed in any preceding claim, in which the path of the gaseous reactant is confined to the surface of the film of liquid reactant.

12. A process as claimed in any preceding

claim, in which the flow of gaseous reactant is concurrent with the liquid reactant.

13. A process as claimed in any preceding claim, in which the liquid reactant is an organic material and the gaseous reactant is sulphur trioxide.

14. A process as claimed in Claim 13, in which the organic material is a hydrocarbon.

15. A process as claimed in Claim 14, in which the hydrocarbon is an alkylbenzene that on sulphonation gives rise to products having surface-active properties.

16. A process for chemically reacting a gas with a liquid substantially as herein described with reference to the accompanying drawing.

17. A process for chemically reacting a gas with a liquid substantially as herein described with reference to the Example.

18. A reaction product when obtained by a process claimed in any preceding claim.

19. Apparatus for chemically reacting a gas with a liquid, which comprises a rotatable disc of a heat-conductive material, means for feeding a liquid reactant to the surface of one side of the disc and adjacent to its centre, so that when the disc is rotated the liquid reactant is spread across that surface in a fast moving thin film by centrifugal forces arising from the motion imparted to the liquid reactant by the frictional drag of the disc; means for feeding a gaseous reactant to the space adjacent to the surface of the disc on which the liquid film is formed for exposure of the liquid film to the gaseous reactant, heat transfer means for removing heat from or supplying heat to the surface of the opposite side of the disc, and means for collecting the reaction product from the periphery of the disc.

20. Apparatus according to Claim 19, in which the means for feeding the liquid reactant to the rotatable disc is arranged for the feeding of liquid over the whole of an

area of the surface of the disc surrounding the centre of the disc.

21. Apparatus according to Claim 19 or Claim 20, in which the means for feeding the gaseous reactant includes a stationary disc arranged parallel with the rotatable disc so as to confine the path of the gaseous reactant to the surface of the film of liquid reactant.

22. Apparatus according to Claim 21, in which the means for feeding the gaseous reactant is disposed for flow of the gaseous reactant concurrent with the film of liquid reactant.

23. Apparatus according to any one of Claims 19 to 22, in which the heat transfer means comprises means for feeding a heat transfer liquid to the surface of the opposite side of the rotatable disc and adjacent to its centre, so that when this disc is rotated the heat transfer liquid is spread across that surface in a fast moving thin film by centrifugal forces arising from the motion imparted to the heat transfer liquid by the frictional drag of the disc.

24. Apparatus according to Claim 23, in which the means for feeding the heat transfer liquid to the rotatable disc is arranged for the feeding of liquid over the whole of an area of the surface of the disc surrounding the centre of the disc.

25. Apparatus according to any preceding claim, in which the means for collecting the reaction product comprise an annular channel at the periphery of the rotating disc and a pipe whose inlet is within the channel.

26. Apparatus for chemically reacting a gas with a liquid substantially as herein described with reference to the accompanying drawing.

UNILEVER LIMITED,
R. Jones,
Agent for the Applicants.

1080863 COMPLETE SPECIFICATION
1 SHEET This drawing is a reproduction of
the Original on a reduced scale

